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STUDY OF ALUMINA-TRICHITE REINFORCEMENT OF A NICKEL-BASED
MATRIX BY MEANS OF POWDER METALLURGY

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16. Abstract Research has been conducted on reinforcing nickel- based matrices with alumina trichites by using powder metallurgy. Alumina trichites previously coated with nickel are magnetically aligned. The felt obtained is then sintered under a light pressure at a temperature just below the melting point of nickel. The halogenated atmosphere technique makes it possible to incorporate a large number of additive elements such as chromium, titanium, zirconium, tantalum, niobium, aluminum, etc. It does not appear that going from laboratory-scale to a semi-industrial scale in production would create any major problems.			
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STUDY OF ALUMINA-TRICHITE REINFORCEMENT OF A NICKEL-BASED MATRIX BY MEANS
OF POWDER METALLURGY

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Research has been conducted for the purpose of reinforcing nickel-based matrices with alumina trichites by using powder metallurgy. Alumina trichites previously coated with nickel are magnetically aligned. The felt obtained is then sintered under a light pressure at a temperature just below the melting point of nickel. Mechanical testing has shown an appreciable reinforcing effect which is, nevertheless, much less than the theoretical maximum. /1*

The matrix of nickel/alumina trichite felts can then be transformed into a nickel-chromium alloy through a halogenated transfer of the chromium. The sintering under pressure is performed at a temperature slightly less than the alloy liquidus. Its mechanical properties are unfortunately relatively poor, which can be partially explained by an attack of the surface of the trichites, revealed through scanning electron microscopy and for which chromium appears to be responsible.

The relatively poor adherence at the trichite/matrix interface and a certain breakage of the fibers during hot compaction also explain the poor mechanical properties.

A technique for aligning the trichites by calendaring has also been tried.

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1. GENERALITIES

Currently, when referring to trichites, we mean monocrystal filaments which are highly perfect crystals. Indeed, the major imperfection and generally the only one is an axial dislocation which is at the origin of the growth of the trichite. In addition, certain imperfect trichites display corner dislocations perpendicular to the axis.

Natural trichites were observed and reported in 1575 by L. Erker (Reference 1) in copper and silver sulphides.

The formation of artificial trichites was discovered in 1940 in an unexpected manner since it was at the origin of short-circuits in miniaturized circuits.

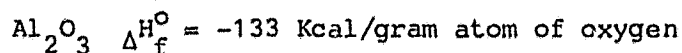
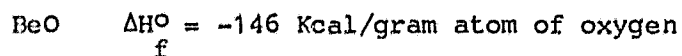
In 1952, Herring and Galt (Reference 2) of Bell Telephone Laboratories showed that the strength of tin trichites is close to that predicted by the theory of perfect crystals. Their high strength is attributed to the perfection of their structure and to their small size which limits the number of defects responsible for the drop in the mechanical properties of the materials when taken in a solid mass.

Since then, a very large assortment of trichites has been prepared. Among others, we can mention: Si, C, Fe, Ni, Ag, Cu, SiO_2 , Au, Zn, Cr, Cd, Sn, B_4C , Al_2O_3 , B, BeO, SiC, Si_3N_4 ... but this list is not limiting and, often, a new type of trichite is prepared.

For the reinforcement of metals that are to be used at high temperatures, the most interesting materials are, of course, those with a high melting point but, in addition, they must absolutely respect a compatibility rule which has, as the primary criterion, an absence of chemical reactivity or

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solubility in the presence of the associated metals. Of the currently known and available trichites, only compounds formed at a high temperature, such as BeO and Al₂O₃, meet this requirement. The Standard formation temperatures are respectively:



We see that beryllium oxide is more stable than alumina but its toxicity properties make it unacceptable, a priori. Moreover, BeO trichites are only manufactured in small quantities, at the laboratory level. On the other hand, alumina trichites are prepared in large quantities by several companies among which we can mention:

- in the United States : Thermokinetic Fibers Inc.

Horizons Incorporated

- in France : Compagnie Française Thomson Houston.

In its sintered form, alumina is a well-known remarkable refractory product. The high temperature at which it is formed gives it an excellent chemical stability and an excellent thermal stability. Its high melting point, 2,050 degrees C, and its mechanical properties retained at high temperatures -indeed, sintered alumina as well as trichites retain at 1200 degrees C 50% of the mechanical strength at normal temperature - make its use possible within a large temperature range.

Moreover, its rather low mass per unit volume, 3.96 g/cm³, is suitable to the manufacture of a compound to be used in an aeronautical application.

But this very interesting material displays a major disadvantage: its highly fragile nature when exposed to thermal and mechanical shocks.

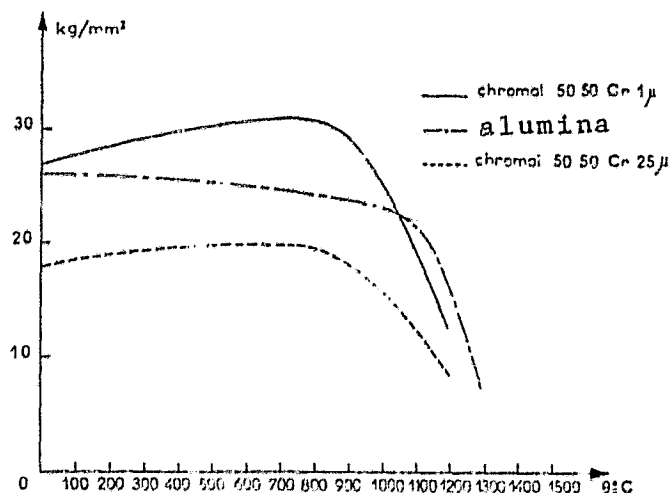


Figure 1 - Tensile Strength as a Function of Temperature

In order to correct these flaws, we thought about bonding it to a metal or an alloy so as to form a metal/ceramic mixture. Research led to the creation of chromium/alumina mixtures in France (Reference 3) and to chromium/molybdenum/alumina mixtures in the United States.

These materials with a ceramic structure thus show a double continuum, metallic on the one hand, ceramic on the other. The mechanical properties are excellent. Figure 1 shows breaking strength curves as a function of temperature for alumina, for a chromium/alumina mixture made with fine chromium with a 1 μ micron grain size, and for an identical mixture except that it was made with electrolytic chromium with a 25 μ micron grain size.

We note that it is only after a temperature of 1,050 degrees C that the alumina/chromium mixture (Cr 1 μ micron) sees its mechanical properties drop below those of alumina and, despite that, at 1,200 degrees C, its breaking strength is still greater than 10 hb.

The thermal shock resistance is good as the presence of the chromium

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provides a good thermal conductivity. Figure 2 shows the test results for temperature differences of ΔT . We have expressed the ratio of the breaking strength of a given material subjected to a thermal shock of ΔT to the breaking strength of the same material that has not undergone the same treatment, as a function of the thermal shock ΔT . Alumina loses its strength quickly and it reaches the zero value at $\Delta T=600^\circ$. The threshold below which no degradation is observed is 100 degrees C for the chromium/alumina mixture whereas it is only 50 degrees C for alumina. Creep resistance is excellent but leads to an unpredictable failure without any appreciable elongation. Finally, resistance to mechanical shock is poor, close to that of alumina.

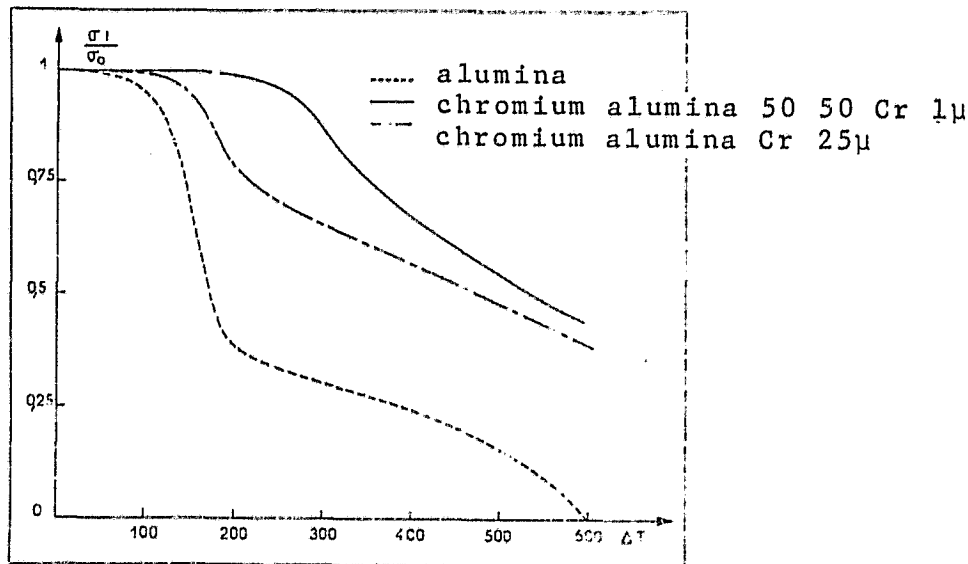


Figure 2 Resistance to Thermal Shock

These last observations led us to abandon this material for applications in turbojet engines.

Reinforcement using trichites excludes the formation of a continuous ceramic phase. From this, we can hope to avoid the mechanical fragility that

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results from the self-propagating nature of a crack that starts in the alumina.

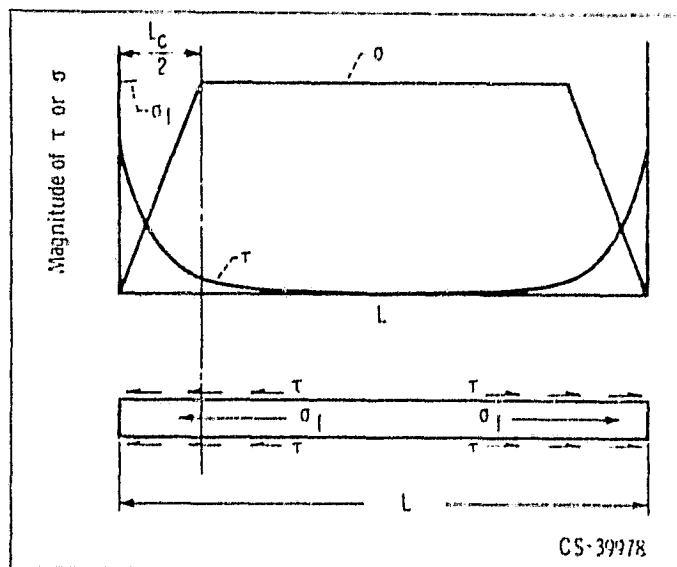
It appears necessary to us to recall beforehand the principles and the conditions for reinforcing a material with fibers. In a general manner, the applied stress is mainly sustained by the fibers. The matrix only keeps the fibers together, transmits the stress from one fiber to another through shear, and protects the surface of the fibers.

For a coated fiber inside a matrix stressed mechanically in the direction of the fiber:

- the shear stress τ is maximum at the ends and equal to zero at the center;
- the tensile stress is equal to zero at the ends and maximum starting at a distance $l_c/2$ from the ends (l_c is the critical length of the fiber).

Figure 3 graphically describes these two facts.

Figure 3. Distribution of the stresses in a composite material with aligned fibers, according to Petrusek



We can mention a certain number of conditions necessary to the reinforcement of a material with fibers (Reference 4).

1. The resistance and the modulus of the fiber must be much higher than

those of the matrix. This is true for the Ni-Al₂O₃ couple.

at 25 degrees C: Ni E = 22,000 hb $\sigma_r = 50$ hb

Al O E = 50,000 hb $\sigma_r = 1,000$ hb

This difference only increases with temperature.

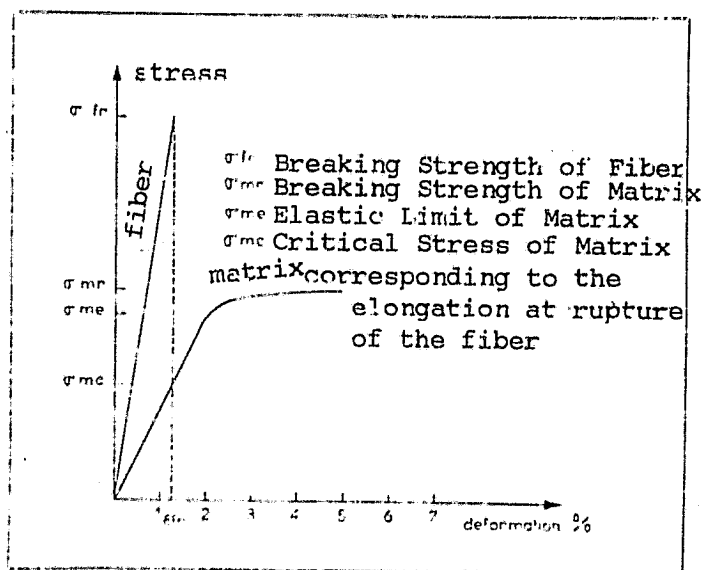
at 1,000 deg. C: Ni E 10,000 hb $\sigma_r = 3$ hb

AL O E = 30,000 hb $\sigma_r = 420$ to 740 hb

2. The volumetric fraction V_F of fibers must be sufficiently high in order to have reinforcement.

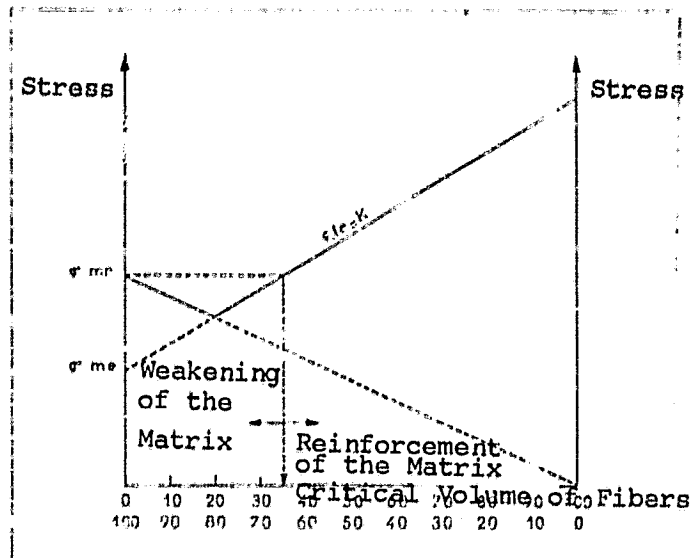
If we show, in the same diagram, the stress-deformation curves for the matrix and the fibers (Figure 4), we then see that when the fibers break for a stress σ_{fr} with a rather low deformation, the matrix breaks for the same deformation, under a stress σ_{mc} less than its maximum breaking strength, at least from a macroscopic standpoint. We can then plot the diagram of Figure 5 which shows the variation of the breaking strength of the composite as a function of the volumetric fraction of the fibers.

Figure 4. Comparison of the stress-deformation diagrams of a ceramic fiber and of a metallic matrix.



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Figure 5. Determination of the critical volume of fibers in a composite according to Richard H. Krock.



Immediately, we see two important points in the diagram: the minimum volume of the fibers which corresponds to a composite having a minimum breaking strength (lower than that of the matrix by itself) and the fiber critical volume which corresponds to the limiting volume beyond which the strength of the composite is greater than that of the matrix alone.

In the case of chromium and alumina, the critical volume is rather low. If we assume the nickel at a normal temperature $\sigma_{mc} = 6 \text{ hb}$ and $\sigma_{mr} = 32 \text{ hb}$, for fibers having $\sigma_F = 350 \text{ hb}$, $V_{crit} = 7.33 \%$ and for fibers having $\sigma_F = 700 \text{ hb}$, $V_{crit} = 3.63\%$.

3. The length of the fibers must be greater than a minimum value below which reinforcement is negligible.

4. The fiber-matrix binding must be solid and resilient. It is necessary for the wetting between the matrix and the fiber to be good.

Papers published on this subject indicates poor wetting of alumina by molten nickel. Research work performed by Sutton (Reference 5) and Ritter (Reference 6) using the molten drop on an alumina monocrystal approach has shown that small additions of chromium, titanium or zirconium to nickel increase the wettability but seriously alter the alumina surface. The

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aggressiveness level increases in the chromium-titanium-zirconium direction.

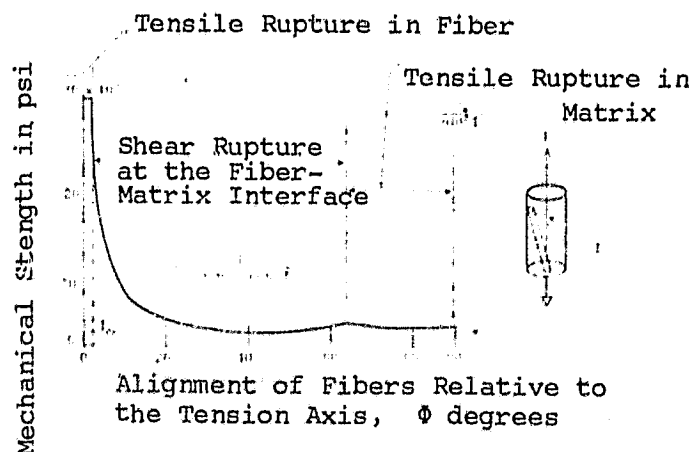
5. The coefficients of expansion must be rather close to each other. For nickel and alumina, we have, respectively, $12 \times 10^{-6}/\text{degree}$ and $6 \times 10^{-6}/\text{degree}$. Therefore, they are appreciably different. Nevertheless, it is certain that it is difficult to find metals with coefficients of expansion close to that of ceramic. Only the refractory metals such as tungsten and molybdenum have this characteristic but they display poor oxidation characteristics.

6. The fiber must be stable at its normal temperature and at a high temperature. It must keep a large portion of its mechanical strength at a high temperature. For alumina, it is true since up to 1,200 degrees Centigrade, it retains 50% of its mechanical properties found at a normal temperature.

7. The fibers must be suitably oriented in one direction to optimize the reinforcement in this direction and must be regularly distributed.

Figure 6 is a curve proposed by Petrasek (Reference 7) which shows the variation in tensile strength of the composite as a function of the misorientation of the fibers.

Figure 6 . Effect of the alignment of the fibers on the strength of a composite according to Petrasek.



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We see that for small misalignments of the fibers, not only the mechanical properties of the composite decrease greatly but the type of rupture changes.

8. An approximate computation makes it possible to predict that, theoretically, with 20% of the volume composed of fibers, and by assuming the contribution by the metal to be negligible to the reinforcement, at 1,100 degrees C, an alumina trichites-nickel composite should provide a breaking point of 125 hb. This potential strength of the metals reinforced by alumina trichites explains the interest created by this type of binding. We will see that we are still far from it.

2. SELECTION OF THE METHOD FOR PREPARING THE ALUMINA TRICHITES-NICKEL COMPOSITE

The major methods that we can envisage to prepare a composite are the following:

- infiltration of a structure of oriented trichites in a liquid phase. This has been used by Sutton (Reference 8) to make-up alumina trichites-silver composites,
- deposit of the metal through decomposition of a volatile compound,
- electrolytic deposit of the metal on trichites oriented by means of a liquid flow or centrifugation ,
- powder metallurgy.

The first two techniques are difficult to implement. The third (electrolytic deposit) has the advantage of coating the trichites without causing their deterioration but the volume of fibers that can be incorporated remains

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low. Moreover, metals such as chromium are hard to add (Reference 9).

Powder metallurgy has already been used by several authors, including Parratt (Reference 10). Metal powder was dispensed with trichites in appropriate liquids. The whole was filtered and then sintered. The trichites are distributed at random but parallel to the plane of filtration. Preferably, it is necessary for the diameter of the powder to be close to that of the diameter of the trichites. Instead of a mixture with a powder, a prior coating of the trichites with the metal should eliminate this requirement and, in addition, in the case of nickel, it should make it possible to have a magnetic alignment. It is by starting with this idea that the experiment has been undertaken.

3. DESCRIPTION OF THE METHOD USED

3.1. Reinforcement Base Material.

These are types of alumina trichites, with the Al_2O_3 formula, that have the following mechanical characteristics:

- tensile strength $\sigma_F = 1,000$ to $1,200$ hb at 25 degrees C,
- elongation at rupture $A = 2\%$ elastic for temperatures lower than $1,000$ degrees C;
- modulus of elasticity $E = 50,000$ hb.

Their length is between 3 and 10 mm and their "diameter" between 0.5 and 10 microns. The cross-section is either hexagonal or parallelepipedal depending on the growth direction.

Figure 7 is a picture of a felt of alumina trichites as delivered, taken with a scanning electron microscope. Figure 8 shows a similar one made

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Figure 7 - Alumina trichites observed with the help of a scanning electron microscope.

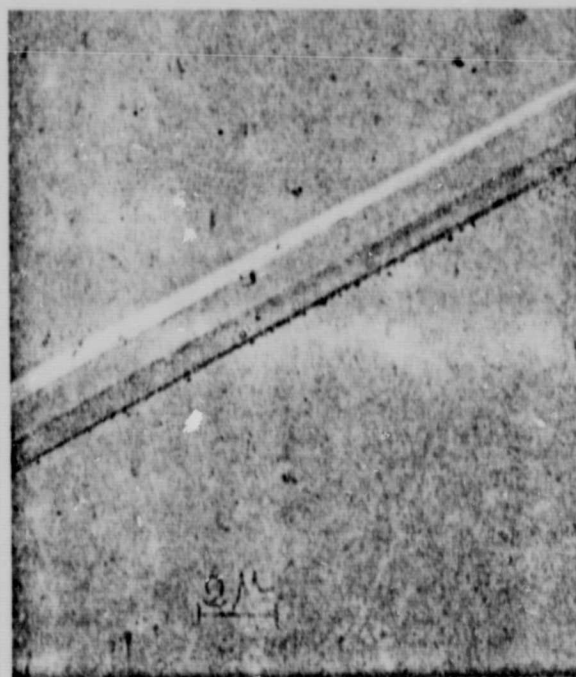


Figure 8 - Alumina trichite with an hexagonal cross-section (similar one made of carbon)

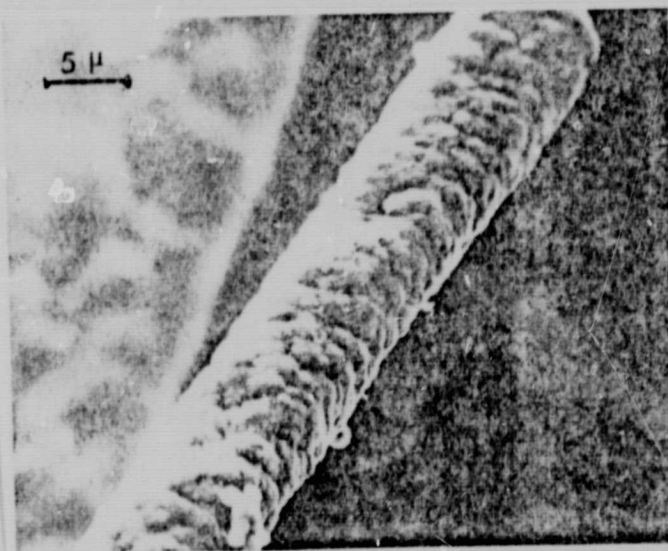
of carbon made with a trichite having a hexagonal cross-section as seen with an electron microscope.

3.2. Nickel-coating the trichites

The Canadian company Sherritt Gordon has perfected a process for coating various particles with nickel or cobalt. The operation takes place at about 200 degrees C inside an autoclave under a hydrogen pressure through reduction of a salt of the metal to be deposited. Alumina is mentioned among the products likely to be coated using this method. We will check it on alumina particles of a poly-crystal nature. Unfortunately, the single-crystal trichites behave much worse, probably because of the reticular perfection of their surfaces. In this case, the nickel deposit is partial and hardly sticks. To overcome this difficulty, we had to resort to a trick. It consisted of previously making a deposit of pyrolytic carbon on the trichite felts; this deposit lends itself to a proper coating and can later be eliminated in moist hydrogen. Figure 9 shows the appearance of the nickel deposit over a trichite (observation made with the help of the scanning electron microscope).

This method makes it possible, to a certain extent and by modifying the thickness of the nickel deposit, to modify the volume of the fibers inside the matrix.

Figure 9 - Nickel-coated alumina trichites (scanning electron microscope)



3.3. Alignment of the Nickel-Coated Trichites

We have seen that, in order to optimize reinforcement, it is necessary to align the fibers in one direction. The trichites being coated with nickel, we have used a magnetic method of alignment. It includes an ultrasonic tank inside which the nickel-coated trichites are maintained in suspension in some ethyl glycol. Using a siphon, they are carried into a sedimentation tank placed inside the air gap of a magnetic circuit. The glycol is recycled into the ultrasonic tank by a pump.

The felt obtained is then dried in an oven then sintered and decarbonized in moist hydrogen. The concentration in residual carbon is about 500 ppm. In that manner, we obtain a rigid structure with high porosity.

3.4. Sintering Under Pressure

Strips measuring 6 mm in width are cut from the felts, stacked in a graphite matrix which has all six faces covered with alumina plates designed to prevent carburization during the high-temperature treatment.

The apparatus for sintering under pressure is composed of an oven, heated by means of HF and operating in a secondary vacuum. The pressing is done at a temperature of 1,430 degrees C, slightly below the melting point of nickel. The pressure applied is about 35 kg/cm².

This operation provides a material with a residual porosity in the order of 5 to 7% and a fiber volume of 18%.

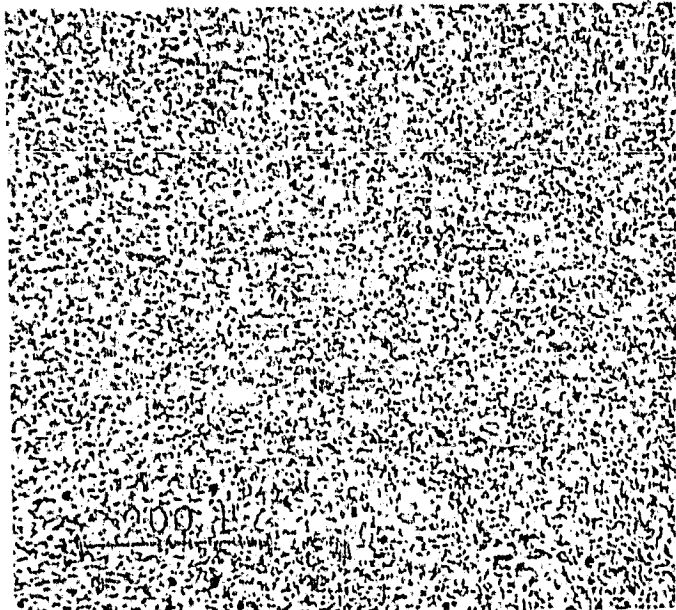
Figures 10 and 11 are, respectively, longitudinal and transversal cross-sections of such a material.

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Figure 10 - Longitudinal
Cross-Section of a Nickel-
Alumina Trichites Composite



Figure 10 - Transversal
Cross-Section of a Nickel-
Alumina Trichites Composite



3.5 - Results

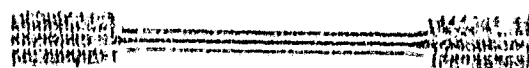
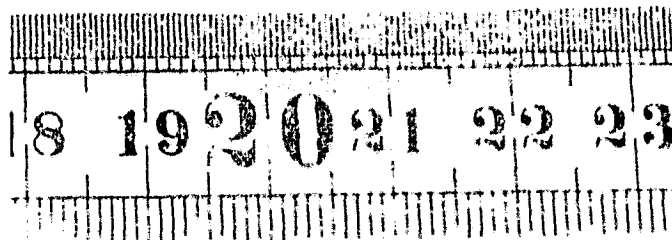
Starting with the rods obtained through compression, we extract tension test specimens with threaded heads. Figure 12 shows one of these test specimens.

Tension tests are conducted on an Adamel machine in a vacuum at several temperatures: 25, 500, 800 and 1,100 degrees Centigrade.

Standard test specimens are prepared from ex-carbonyl nickel sintered using the same technique.

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Figure 12 - Tension Test Specimen.



In a general sense, we can say that:

- tension strength of the composite is slightly less than that of nickel at normal temperature for an elongation at rupture of about 1%
- tension strength of the composite is about twice that of nickel at 1,100 degrees C. Elongation at rupture is from 8 to 11 %.

Figure 13 shows the stress-deformation curves for nickel and for the nickel-alumina trichite composite at a temperature of 1,100 degrees C.

Figure 13 - Stress-Deformation Curves for pure nickel and for a nickel-alumina trichite composite at 1,100 degrees C.

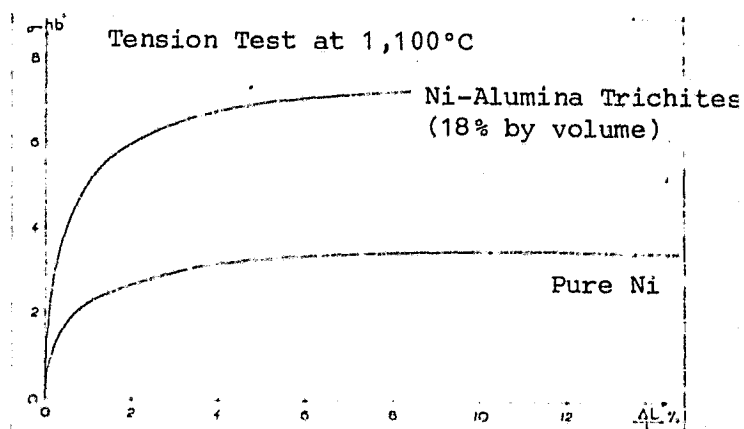
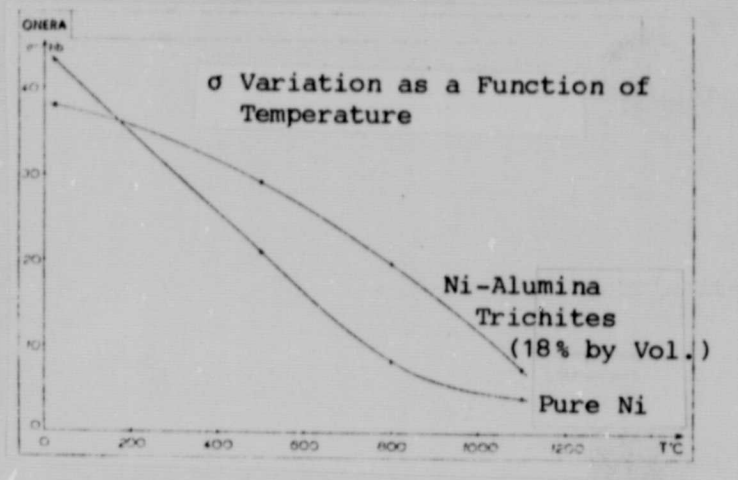


Figure 14 depicts the variation of the breaking strength as a function of temperature for nickel and for the composite.

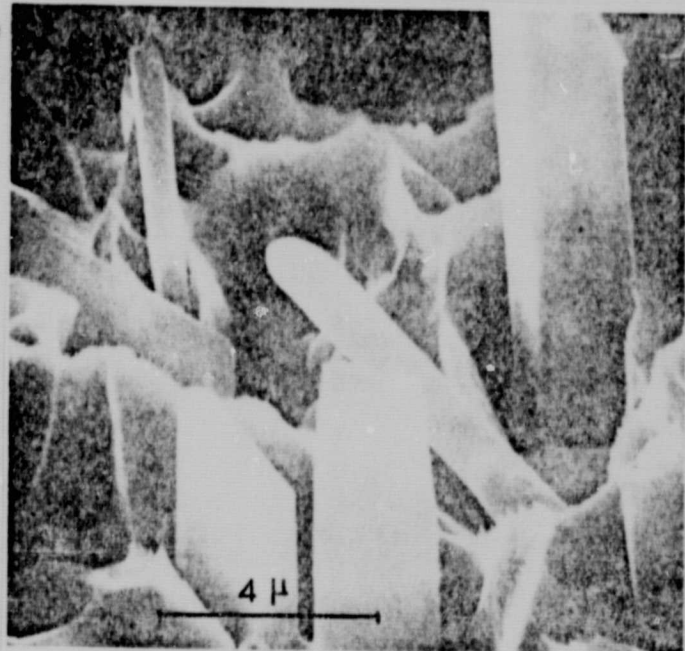
Figure 14 - Variation of the yield strength as a function of temperature for nickel and for the nickel-alumina trichites composite.



The composite has a breaking strength that is less than that of nickel at low temperatures. This is not well explained and may be due to phenomena associated with interfacial bonding or to fibers breaking.

Photographs of the fracture have been taken with a scanning electron microscope and Figure 15 shows trichites coming out of the matrix.

Figure 15 - Photograph of the fracture of a composite material (scanning electron microscope)



3.6 - Other Method for Aligning Trichites, by Calendering

This method avoids pre-coating the trichites with the metal used in the matrix. In addition, it makes it possible to align trichites in a metal that we cannot deposit on the fibers or that we can deposit but that is not ferromagnetic.

Trichites and powder from the metal are scattered inside a plastic and the whole is calendered. The plastic is then eliminated. The manufacturing process is then the same as before.

Mechanical tests have given a breaking strength of 7 hb at 1,100 deg. C, but a fragility in the material appeared and could not be explained. In Figure 16, we see the trichites aligned using this technique inside a nickel matrix.

Figure 16 - Trichites Aligned
Through Calendering



4. MAKING A CHROMIUM 80-20/NICKEL MATRIX

Chromium is needed to give the material a good resistance to oxidation. It is also needed to increase the adherence qualities at the matrix/fiber

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interface (work by Sutton, Ritter). They have shown that nickel with 1% of chromium added sticks well to an alumina monocrystal. With 8% chromium, it is no longer possible to detach the metallic drop and, sometimes, we split the alumina crystal. It is possible that this good bonding is done through a lesion of the crystal of the surface. This is not recommended for trichites.

To make a chromium 80/20-nickel matrix, we have followed the same path as for the nickel matrix:

- nickel-coating the trichites
- magnetic alignment
- decarburization, primary sintering.

The felts obtained are then chromated by immersion in a porous granulated mixture of nickel and chromium in a suitable proportion. The transfer of chromium takes place at about 900 degrees C by means of a halogenated gaseous phase.

Hot-pressing takes place in a vacuum at a temperature of 1,380 degrees C, slightly less than the solidus of the alloy (1,400 degrees C).

Results of mechanical tests are rather disappointing. We have found 5.3hb at 1,100 degrees C.

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Observations performed with scanning electron microscopy have shown etching of the surface of the trichites. Pressing tests at temperatures lower than 1,380 degrees C have also revealed an attack that decreases as a function of temperature but that still exists at 1,100 degrees C.

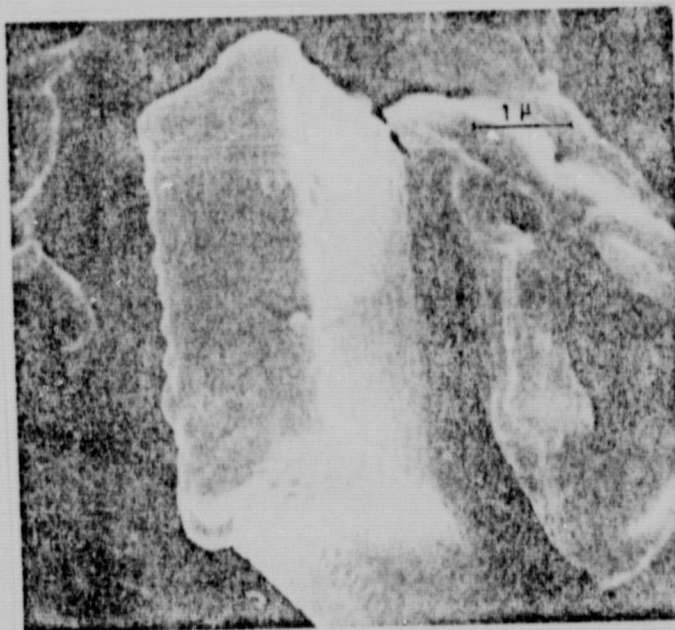
Figures 17, 18 and 19 show the appearance of the surface of the trichites, observed with scanning electron microscopy, for composites subjected, respectively, to temperatures of 1,100, 1,200 and 1,250 degrees C. We

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Figure 17 - Appearance of the
Surface of a Trichite Subjected
to Heat at 1,100 degrees C in a
Nickel-Chromium Matrix (scanning
electron microscope)



Figure 18 - Appearance of the
Surface of a Trichite Subjected
to Heat at 1,200 degrees C in a
Nickel-Chromium Matrix (scanning
electron microscope)



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Figure 19 - Appearance of the Surface of a Trichite Subjected to Heat at 1,250 degrees C in a Nickel-Chromium Matrix (scanning electron microscope)



already notice an attack at 1,100 degrees C. This partly explains the poor mechanical properties and it is likely that chromium is responsible for this etching. It would appear necessary to consider protecting the trichites with a layer that would prevent the chromium from reaching the trichite but that would be advantageous from the standpoint of adherence at the interface.

Identical observations have been made with alumina trichites that had been coated with titanium before being incorporated into a nickel matrix by electrolytic means (work by M. Marchal at Compagnie Française Thomson-Houston). Figures 20 and 21 are views, taken with scanning electron microscopy, of the surface of such trichites inside a composite heated to 800 degrees C. We note the analogy between the attacks attributable to chromium and titanium and we observe that for an approximately equivalent etching, the temperature is lower for titanium. All of these facts confirm the observations made by Sutton, performed on an alumina

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Figure 20 - Appearance of the Surface of a Trichite Pre-Coated with Titanium Heated to 800 deg. C in a nickel-alumina trichite composite (scanning electron scope)

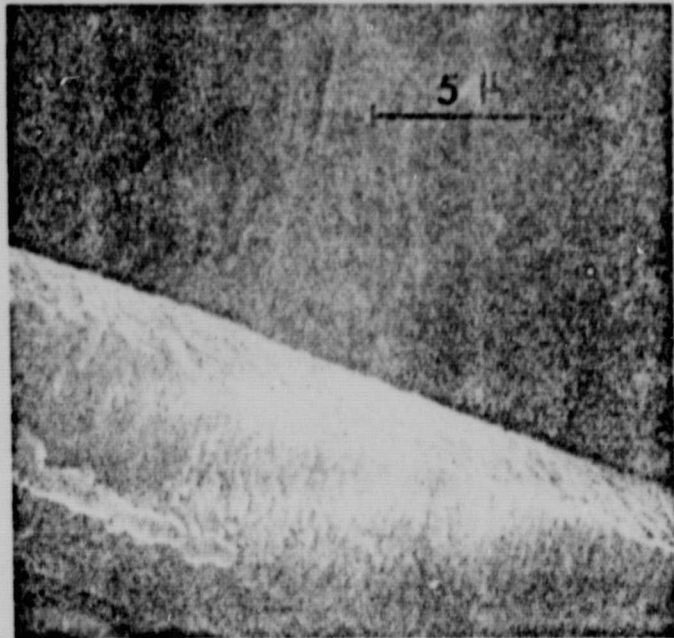
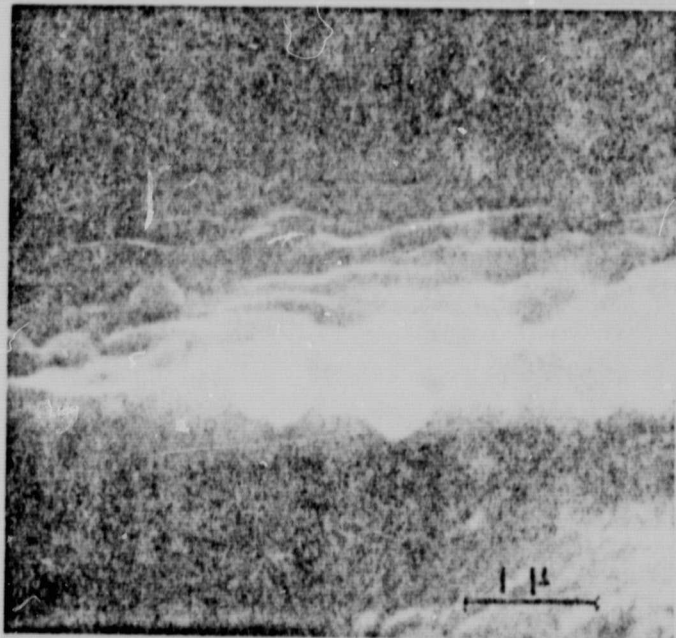


Figure 21 - Same Observation as For Figure 20 on Another Area of the Break



monocrystal during studies on adherence. Sutton has also suggested the creation of a carbide layer of titanium, vanadium or zirconium to protect and wet the surface of the alumina trichites.

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CONCLUSION

To obtain composite materials composed of a metallic nickel-based matrix reinforced with alumina trichites, a procedure has been imagined and perfected. There are several advantages.

It can be transferred to cobalt without difficulties. Thanks to the halogenated atmosphere technique, it makes it possible to incorporate a large number of additive elements such as chromium, titanium, zirconium, tantalum, niobium, aluminum, etc.. It does not appear that going from laboratory-scale to a semi-industrial scale in production would create any major problems.

On the other hand, the process presents certain disadvantages.

If we wish to avoid high pressures, sintering under pressure has to take place in the neighborhood of the melting point of the alloy and it facilitates reactions between alumina and certain additive elements.

During this operation, the one-dimensional shrinkage is very important and risks causing the breaking of trichites, hence decreasing their effectiveness.

With respect to mechanical tests, they show that the high temperature strength that can be theoretically obtained is far from being reached.

Examinations using scanning electron microscopy have shown that acquiring the optimum properties of the material encounters two contradictory limitations.

They are: either insufficient adherence at the interface which does not make it possible to transfer the load entirely to the fibers; or, if this flaw is corrected by an appropriate additive, an alteration of the surfaces of the trichites causing their weakening.

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The first disadvantage has been encountered with the pure nickel matrix; the second with the nickel-chromium or nickel-titanium matrixes. Between the two stumbling blocks, the maneuvering range of the research is particularly restricted.

However, we can conceive several approaches for solving the problem, such as interposing an intermediate layer. However, we must not conceal that major research work, both technological and basic, will be necessary. Under these conditions, the perspectives for the applications of a material of this type are not within reach.

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